

# POTASSIUM CORROSION TEST LOOP DEVELOPMENT

Quarterly Progress Report No.10 For Quarter Ending January 15, 1966

EDITED BY E.E. HOFFMAN

prepared for NATIONAL AERONAUTICS AND SPACE ADMINISTRATION Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
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CINCINNATI, OHIO 45215

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QUARTERLY PROGRESS REPORT NO. 10

Covering the Period

October 15, 1965 to January 15, 1966

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Lewis Research Center

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#### POTASSIUM CORROSION TEST LOOP DEVELOPMENT

#### I. INTRODUCTION

This report covers the period, from October 15, 1965 to January 15, 1966, of a program to develop a Prototype Corrosion Test Loop for the evaluation of refractory alloys in boiling and condensing potassium environments which simulate projected space electric power systems. The prototype test consists of a two-loop Cb-1Zr facility; sodium is heated by direct resistance in the primary loop and used in a heat exchanger to boil potassium in the secondary corrosion test loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high-vacuum environment. The immediate corrosion test design conditions are shown below; it is expected that the temperature could be increased by about 400°F when testing is extended to include refractory alloys stronger than Cb-1Zr.

- 1. Boiling temperature, 1900°F
- 2. Superheat temperature, 2000°F
- 3. Condensing temperature, 1350°F
- 4. Subcooling temperature, 800°F
- 5. Mass flow rate, 20 to 40 lb/hr
- 6. Vapor velocity, 100 to 150 ft/sec
- 7. Average heat flux in the potassium boiler 50,000 to 100,000 BTU/hr ft<sup>2</sup>

The development program includes the construction and operation of three Cb-1Zr test loops, each of which are being used in a sequence of component evaluation and endurance testing. Loop I, a natural convection loop, has been operated for 1,000 hours with liquid sodium at a maximum temperature of 2260°F to 2380°F to evaluate the electrical power vacuum feedthroughs, thermocouples, the method of attaching the electrodes, the electrical resistivity characteristics of the heater segment, and the use of thermal and electrical insulation. Loop II, a single-phase sodium, forced-circulation loop to evaluate the primary

loop EM pump, a flowmeter, flow control and isolation valves, and pressure transducers, has completed 2,650 hours of scheduled testing. This loop was operated at a maximum temperature of 2065°F and a pump inlet temperature of 1985°F. The Prototype Corrosion Test Loop, a two-loop system, includes a boiler, turbine simulator and condenser in addition to the above components. This facility will be used to develop and endurance test (5,000 hours) the components required to achieve stable operation at the corrosion test design conditions.

The progress reports and the topical reports which have been issued on this program are listed at the rear of this report. Additional topical reports will be published which cover the alkali metal purification and handling, and the design, operation and evaluation of the Component Evaluation Loop tests and the Prototype Corrosion Loop Test.

#### II. SUMMARY OF PROGRESS

An additional 2,024 hours of test operation of the Prototype Corrosion Loop was completed during the past quarter. A total of 3,809 hours of the 5,000-hour test was completed at 0800 on January 8, 1966 when a pressure rise in the test chamber necessitated a test shutdown. The pressure rise resulted from stress-corrosion cracking of one of the stainless steel water cooling channels of the test chamber wall. The problem has been solved by isolating the "cracked" channel, sealing the cracks and pumping on this channel with an independent vacuum system. The loop will be restored to the test conditions on or about January 20, 1966.

During operation the loop continued to perform in an extremely stable manner with no significant short time changes or long time drift in pressures, temperatures or flow. No adjustments in the metering valve to alter the pressure drop between the potassium pump and the preheater were required. In addition, no significant change in the pressure drop across the metering valve, preheater and boiler was detected.

The pressure in the vacuum chamber has decreased from 2.8 x  $10^{-8}$  to 2.1 x  $10^{-8}$  torr during the last 2,024 hours of the test.

#### III. PROGRAM STATUS

#### A. PROTOTYPE CORROSION LOOP OPERATION

The Prototype Corrosion Loop completed an additional 2,024 hours of operation during the last quarter. As of January 15, 1966, 3,809 hours of test operation had been completed.

Loop operation during this period continued to be extremely stable with no significant fluctuations in pressure, temperature or flow in the two-phase potassium circuit during normal operation. The most important test conditions are summarized in Figure 1 which is data taken after 2,200 hours of operation and is typical of the loop conditions during the entire test as no significant drift or change of any of the temperatures or flow in either the sodium or potassium circuits has been detected. An analysis of the boiler performance after 259 hours of operation was given in the previous progress report (1) and the performance has not changed significantly during the subsequent 3,550 hours of operation. Several minor "incidents" which affected loop operation for brief periods occurred during the last quarter and these events are discussed below. An extended discussion of the test shutdown on January 8, 1966 which was necessitated by a water-to-vacuum leak in one of the test chamber cooling channels will be given in Section B of this report.

On October 16, 1965 at 0800, the electrical power to the test area was interrupted momentarily as the result of a transformer changeover at the main power sub-station of the plant. The drop in line voltage was sufficient to activate the loop safety circuits which turn off power to the loop heaters and EM pumps. The power was turned back on immediately and the loop returned to its steady state test conditions within 30 minutes. Manual re-start is desirable in restoring power to loop circuits following activation of the safety circuits in order to avoid large current surges to the loop heaters and to restore stable loop operation in the minimum of time.

Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 9
for Period Ending October 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54912,
p. 37.

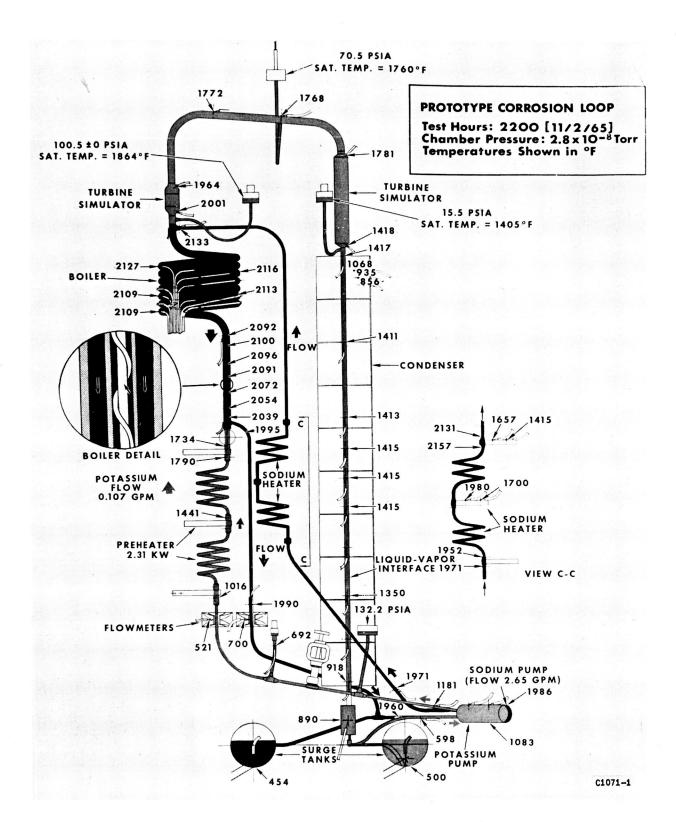


Figure 1. Prototype Corrosion Loop Test Conditions. (CDC4506)

Fluctuations in the getter-ion pump current were recorded on November 19 for approximately 6 hours. These fluctuations were attributed to the formation of a titanium "flake" or "whisker" which resulted in intermittent shorting out of the anode of the ion pump element. This problem did not re-occur during the remainder of the reporting period.

A momentary loss of electrical power to the test area on November 26, during an electrical storm, resulted in the activation of the safety circuits of the loop test system. Power to the loop heaters and EM pumps was restored by the operator on duty and the loop was back to the steady state test conditions in less than five minutes. During the incident the potassium boiler outlet temperature dropped from the normal 2000°F to a minimum of 1980°F.

During the quarter no adjustment of the metering valve was made. This valve is located between the potassium pump outlet and the preheater inlet. A discussion of changes and adjustments of the metering valve during the first 1,785 hours of loop operation was included in the previous quarterly report. During the test period from 1,785 hours to 3,809 hours of loop operation, the pump outlet pressure and the total pressure drop across the metering valve, preheater and boiler have not varied significantly. The pump outlet pressure has been in the range 130.5 to 134.0 psia and the pressure drop across the valve, preheater and boiler in the range 29.7 to 36.7 psi during this period. There has been no trend in the pressure drop which would suggest a build up of a restriction in the flow annulus around the metering valve plug, which has a minimum cross sectional area of 0.002 in<sup>2</sup>.

The pressure in the test chamber decreased from  $2.8 \times 10^{-8}$  to  $2.1 \times 10^{-8}$  to reducing the 2,024 hours of loop operation which was completed in the last quarter. No significant change in the relative concentrations of the individual gaseous species has occurred during the reporting period. Argon (m/e-40) and nitrogen/carbon dioxide (m/e-28) continue to constitute the major portion of the residual gases.

Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 9
for Period Ending October 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54912,
p. 22.

The argon instabilities in the getter-ion vacuum pump which cause momentary pressure excursions in the test chamber pressure continue to occur at fairly regular time intervals as shown in Table I. The instabilities continue to occur every 40 to 70 hours, and no trend in the frequency of the instabilities is apparent. As long as the argon pressure surges do not exceed peak values in the  $10^{-6}$  torr range, they are of no particular significance since the argon flooding system on the chamber will only be activated at a pressure in excess of  $4 \times 10^{-4}$  torr.

#### B. TEST SHUTDOWN

At 0800 on January 8, 1966, the Prototype Loop Test was shutdown after 3,809 hours of continuous operation because of a pressure rise in the vacuum chamber. The pressure rose rapidly from the normal level of 2.1 x 10<sup>-8</sup> torr to 1.0 x 10<sup>-6</sup> torr in 17 minutes. Since the pressure trace gave no indication of being an argon instability, as first suspected, the test engineer gave instructions to shutdown the test. The test chamber pressure and the temperatures of several of the hottest components during the shutdown period are given in Table II. In view of the rapid drop in the temperature of the system, the protective wrapping of Cb-1Zr insulating foil on the loop components, and the chamber pressure levels during the shutdown period, it is concluded that no significant contamination of loop components occurred during this period. The partial pressure analysis system on the chamber aided in locating the source of the pressure rise as increases in the water vapor peak (mass/charge-18) were noted following the test shutdown.

A complete description of the test shutdown and the examinations which were performed to determine the nature and extent of the cracking of the test chamber water cooling channels is given in the Appendix Section of this report. Following repair of the cracked cooling channel the loop test is scheduled to be restarted on or about January 20.

TABLE I. PRESSURE SURGES IN THE PROTOTYPE LOOP TEST CHAMBER
RESULTING FROM ARGON INSTABILITIES IN THE GETTER-ION PUMP

Hours of			Pressure	, Torr
Loop			Base	Peak
<u>Operation</u>	Date	Time	Pressure	Pressure
1840	10-18-65	0705	$3.0 \times 10^{-8}$	$1.5 \times 10^{-6}$
1913	10-21-65	0815	$2.8 \times 10^{-8}$	1.8 x 10 -6
2037	10-26-65	1130	$2.7 \times 10^{-8}$	$5.0 \times 10^{-6}$
2090	10-28-65	1710	$2.6 \times 10^{-8}$	$1.5 \times 10^{-6}$
2141	10-30-65	1930	$2.7 \times 10^{-8}$	$1.5 \times 10^{-7}$
2183	11-1-65	1405	$2.7 \times 10^{-8}$	$9.0 \times 10^{-1}$
2260	11-4-65	1830	$2.6 \times 10^{-8}$	$1.5 \times 10^{-6}$
2325	11-7-65	1222	$2.5 \times 10^{-8}$	$1.5 \times 10^{-6}$
2373	11-9-65	1130	$2.5 \times 10^{-8}$	$1.3 \times 10^{-6}$
2424	11-11-65	1515	$2.5 \times 10^{-8}$	$1.3 \times 10^{-6}$
2478	11-13-65	2105	$2.6 \times 10^{-8}$	$1.3 \times 10^{-6}$
2552	11-16-65	2240	$2.4 \times 10^{-8}$	$1.5 \times 10^{-6}$
2600	11-18-65	2250	$2.6 \times 10^{-8}$	$1.5 \times 10^{-6}$
2653	11-21-65	0355	$2.4 \times 10^{-8}$	$9.0 \times 10^{-7}$
2727	11-24-65	0535	$2.6 \times 10^{-8}$	$1.5 \times 10^{-6}$
2748	11-25-65	1315	$2.5 \times 10^{-8}$	1.0 x 10 <sup>-6</sup>
2848	11-29-65	0915	$2.3 \times 10^{-8}$	1.8 x 10 <sup>-6</sup>
2919	12-2-65	0720	$2.5 \times 10^{-8}$	1.3 x 10 -6
2975	12-4-65	1345	$2.8 \times 10^{-8}$	1.5 x 10 <sup>-6</sup>
3040	12-7-65	0628	$2.5 \times 10^{-8}$	$1.5 \times 10^{\circ}$
3119	12-10-65	1530	$2.4 \times 10^{-8}$	$1.3 \times 10^{-6}$
3175	12-12-65	2130	$2.5 \times 10^{-8}$	$1.5 \times 10^{-6}$
3239	12-15-65	1330	$2.5 \times 10^{-8}$	$1.5 \times 10^{-6}$
3299	12-18-65	0150	$2.5 \times 10^{-8}$	$1.5 \times 10^{-6}$
3368	12-20-65	2245	$2.5 \times 10^{-8}$	$1.5 \times 10^{-6}$
3472	12-25-65	0645	2.5 x 10 <sup>-8</sup>	1.5 x 10 -6
3539	12-28-65	0145	2.4 x 10 <sup>-8</sup>	1.6 x 10 6
3603	12-30-65	1820	$2.3 \times 10^{-8}$	$1.5 \times 10^{-6}$
3661	1-2-66	0410	$2.3 \times 10^{-8}$	1.6 x 10 6
3719	1-4-66	1400	$2.3 \times 10^{-8}$	$1.5 \times 10^{-6}$
3785	1-7-66	0825	2.2 x 10 <sup>-8</sup>	1.3 x 10 <sup>-6</sup>

<sup>\*</sup> Pressure change indicated by getter-ion pump recorder. Base pressure corrected to value indicated by calibrated nude ion gauge on the chamber.

TABLE II. TEST CHAMBER PRESSURE AND THE TEMPERATURE OF THE HOTTEST

PROTOTYPE CORROSION LOOP COMPONENTS DURING THE TEST

SHUTDOWN PERIOD ON JANUARY 8, 1966

Time	Pressure Torr	Na Loop- (1) Max. Temp.	K Loop- (2) Max. Temp.	K Loop- Next Highest Temp.(3) °F
0758	2.1 x 10 <sup>-8</sup>	2135	2000	1975
0800	7.0 x 10 <sup>-8</sup>	2135	2000	1975
0805	2.0 x 10 <sup>-7</sup>	2135	2000	1975
0810	$5.0 \times 10^{-7}$	2135	2000	1975
0815 <sup>(4)</sup>	$1.0 \times 10^{-6}$	2135	2000	1975
0820	$5.0 \times 10^{-5}$	1650	1655	1600
0825	4.0 x 10 <sup>-5</sup>	1600	1490	13 <b>7</b> 5
0830	$4.0 \times 10^{-5}$	1550	1350	1230
0845	$4.5 \times 10^{-5}$	1330	1300	1100
0900	$7.0 \times 10^{-5}$	1190	1100	850
2000				

0900 to Pressure held in  $10^{-5}$  torr range until loop cooled to less than  $300^{\circ}\text{F}$ . 1400

<sup>(1)</sup> Heater exit.

<sup>(2)</sup> Boiler outlet.

<sup>(3)</sup> Turbine simulator (Stage No. 1) outlet.

<sup>(4)</sup> Loop heaters and EM pumps turned off.

## IV. FUTURE PLANS

- A. The Prototype Corrosion Loop will be restored to the operating conditions on or about January 20, 1966.
- B. Preparation of topical reports covering the various portions of the program which have been completed will continue.

#### V. APPENDIX

### A. STRESS-CORROSION CRACKING OF VACUUM CHAMBER COOLING CHANNEL

- 1. Description of Vacuum Chamber. The failure which will be described in this appendix occurred in a water cooling channel of the 4' x 11' vacuum chamber which is being used in a 5,000-hour test to evaluate the compatibility of the refractory alloys, Cb-1Zr and Mo-TZM, with the alkali metals, sodium and potassium. The vacuum chamber which is constructed of Type 304L SS, is shown in Figure 2. Heat radiated from the Prototype Corrosion Loop to the chamber wall is removed by water flowing through water channels located on the chamber walls.
- 2. Test Shutdown Caused by Failure of Chamber Wall and Resultant

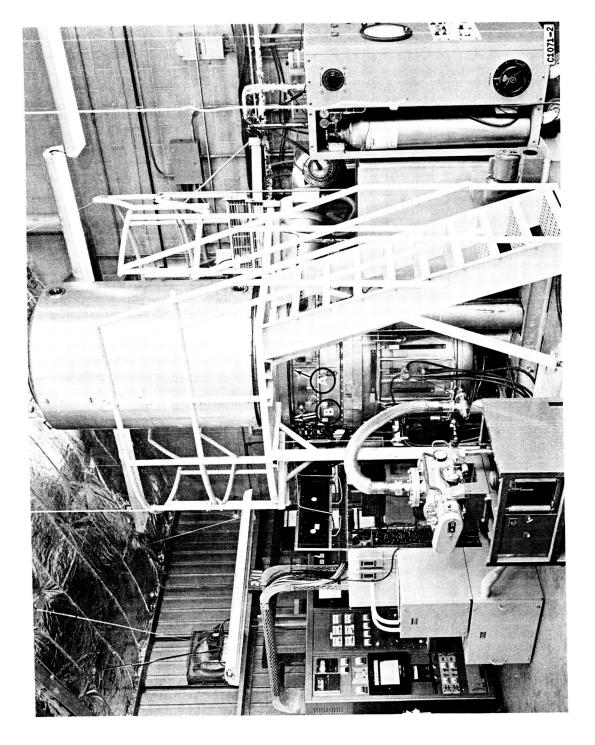
  Pressure Rise. At approximately 0800 on January 8, 1966, the test was shutdown after 3,809 hours of continuous operation because of a pressure rise in
  the vacuum chamber. As shown in Figure 3, the pressure rose rapidly from the
  normal level of  $2.1 \times 10^{-8}$  torr to approximately  $1 \times 10^{-6}$  torr in 17 minutes.

  Since the pressure trace gave no indication of being an argon instability, as
  first suspected, the test engineer gave instructions to shutdown the test.

Partial pressure scans taken after the test loop had been shutdown indicated high concentrations of water vapor and subsequent leak checking established that leaks were present between the water channel in the spool piece and the vacuum side in addition to numerous leaks from the water side of the cooling channel to the air side.

3. Description of Spool Piece, Examinations and Evaluation of the Cooling Channel Failure. A sketch of the spool piece is shown in Figure 4. The water channels on this component and the direction of the water flow are indicated. A cross section of a typical channel is also illustrated. During the leak checking operations it became apparent that a large water-to-vacuum leak was present in a region where the top cooling channel of the spool crossed a vertical weld in the chamber wall. The channel in this region was cut away as shown in Figure 5 and an attempt was made to repair a visible crack in the chamber wall by welding. This only resulted in the opening up of a multitude

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Prototype Corrosion Loop Test Chamber and Associated Equipment. Water Channel in Which Stress-Corrosion Cracking Occurred is Indicated by Arrows. (C65080602) Arrows. Figure 2.

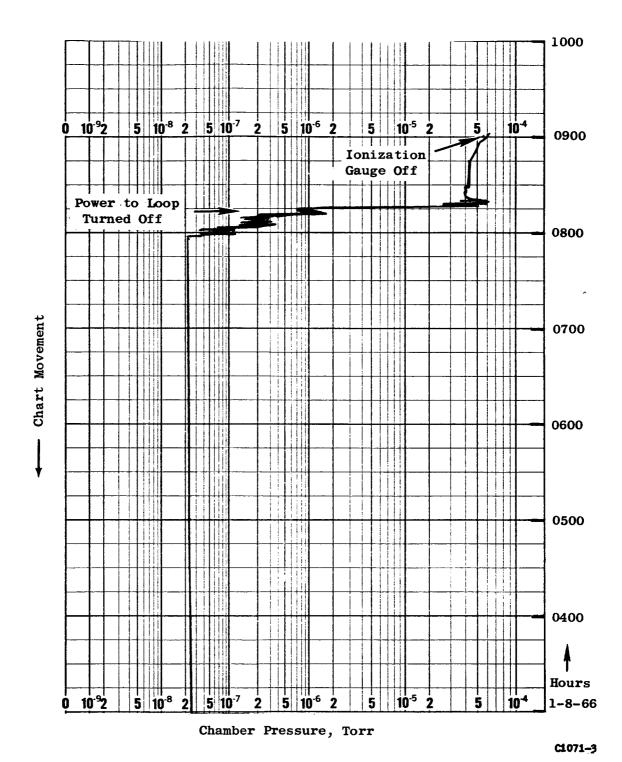
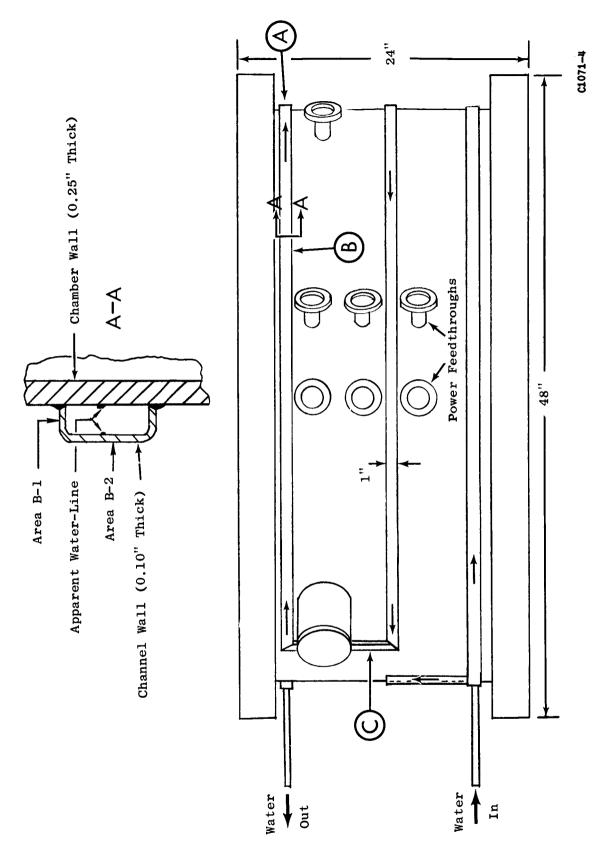


Figure 3. Prototype Loop Vacuum Chamber Pressure During Period When the Test was Shutdown Because of a Water-to-Vacuum Leak in the Top Cooling Channel of the Spool Piece.



Spool Section of Vacuum Chamber Showing Locations, A, B, and C, Where Portions of the Cooling Channel Were Removed. Gross Section of Top Channel Shows Location of Metallographic Samples, B-1 and B-2, and Location of the Water-Line Deposits. Figure 4.

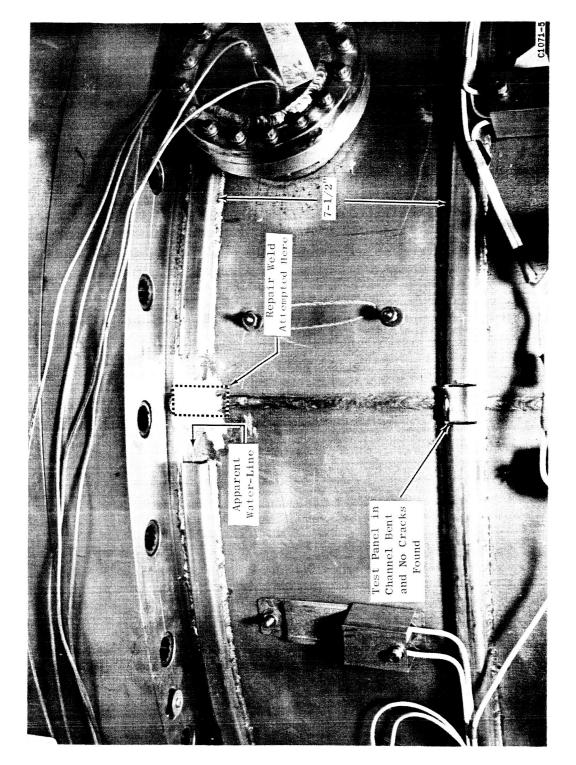


Figure 5. Upper Portion of the Spool Piece of the Chamber Corresponding to Area (A) in Figure 2 Showing Where First Section of Cracked Water Channel was Removed. Lower Channel was Found to be Ductile and Free of Cracks. (C66011401)

of cracks in adjacent portions of the chamber wall. At this point corrosion of some type was suspected and another section of top channel approximately 10 inches long was removed for additional inspection. This area is shown in Figure 6. Examination of the section of removed channel indicated gross cracking of the 0.1-inch thick channel as shown in Figure 7. Figure 7(a) shows the inside surface of the top portion of the channel. Numerous marks made by the grinder used to remove the channel are evident. The complete destruction of the channel wall is apparent in Figure 7(b) which is an enlarged view of a segment of the channel following slight bending. Visual inspection of the chamber wall proper also indicated a multitude of cracks in the upper portion of the channel. Close examination of the cutaway regions of the chamber wall shown in Figure: 5 and 6 revealed a thin line of white deposit near the mid-height of the channel. A corresponding line was found on the segment of channel which had been removed. A portion of this channel was bent so that equal amounts of deformation were given to regions above and below the deposit line. This specimen is shown in Figure 8. The line of white deposits and cracking observed in the region above this line are shown in this photograph. No cracks were detected in the deformed segment of channel located below the deposit line.

Metallographic examination of cross sections of the channel from the lower, uncracked region and the upper, cracked region are shown in Figure 9. (The hardness impressions shown are discussed later in this report.) Additional photomicrographs of the cracked regions are shown in Figure 10 at higher magnifications. The transgranular nature of the cracks are typical of stress-corrosion cracking in austenitic stainless steels (3).

4. Additional Test Results. In an effort to shed as much light as possible on the conditions responsible for the water channel failure described above, a number of tests were conducted and these results are described below.

The results of analyses performed on a sample of the vacuum chamber cooling water are given in Table III. The chloride concentration is thought to be

Phelps, E. H., and Mears, R. B., "The Effect of Composition and Structure of Stainless Steel Upon Resistance to Stress-Corrosion Cracking", First International Congress on Metallic Corrosion, London, April 1961, Butterworths (1962), p. 325.

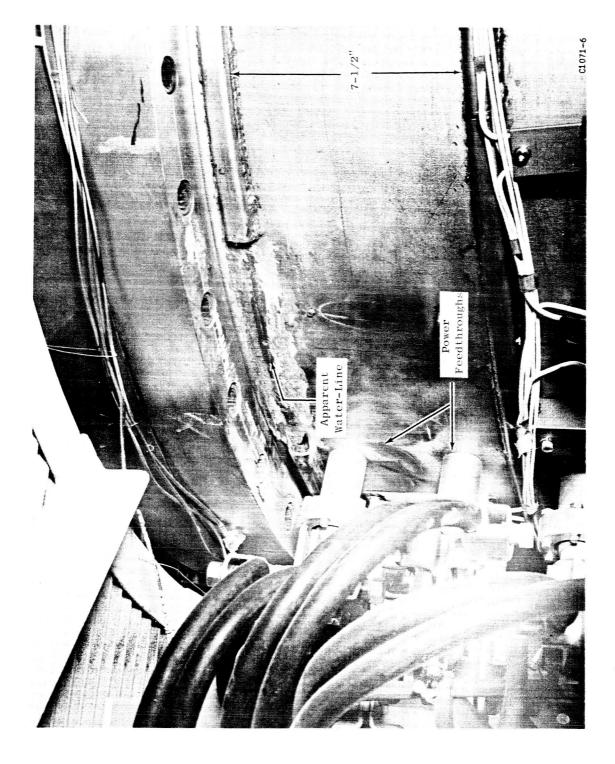
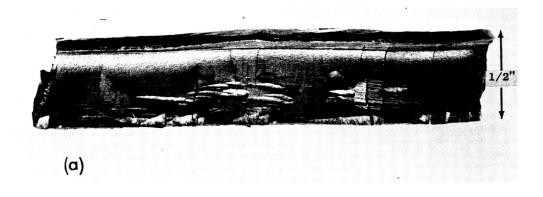


Figure 6. Upper Portion of the Spool Piece of the Chamber Corresponding to Area (B) in Figure 2 Showing Where Section of Cracked Water Channel was Removed. (C66011403)



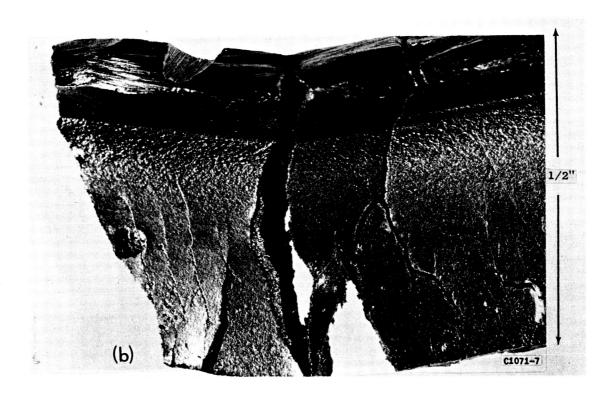


Figure 7. Water Side of the Top Portion of the Upper Cooling Channel of the Chamber Spool Piece. Section Shown was Taken from Area B-1 Indicated in Figure 4. Sample Shown in (b) is Typical of the Region Shown in (a) After Slight Bending. Grinding Marks on the Surface Shown in (a) Occurred During Removal of the Channel.

a) C66012125; b) C66011802

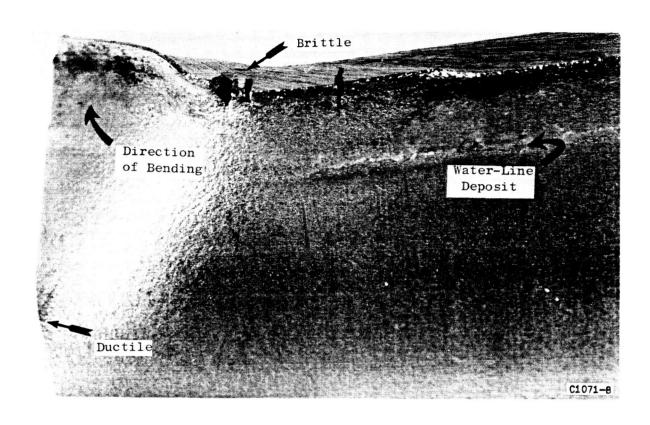
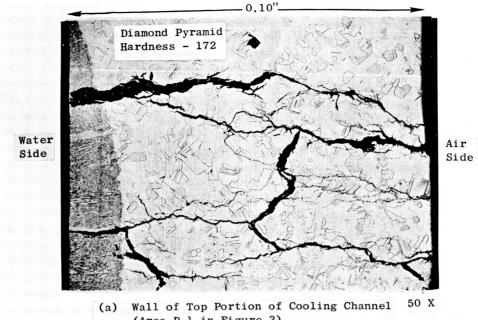
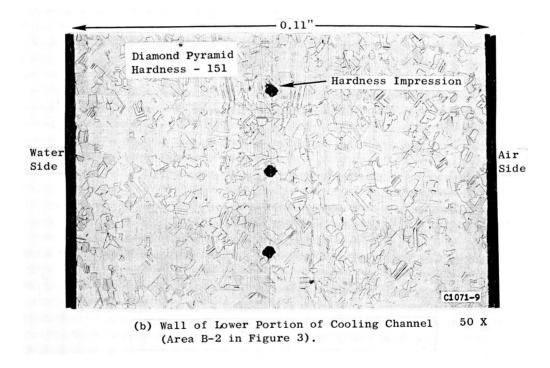


Figure 8. Appearance of Water Side of a Specimen Cut From the Region of the Top Cooling Channel Nea: the Water-Line Deposit. Corner of the Specimen was Bent to Illustrate Brittleness of the 0.10-Inch Thick Channel Above the Deposit Line. (C66011803)



(Area B-1 in Figure 3).



Metallographic Sections of the Top Portion (a) and Lower Portion (b) of the Figure 9. Upper Water Cooling Channel of the Chamber Spool Piece.

Etchant: 10g Oxalic Acid, 100cc Water, Electrolytic

- a) B290118
- b) B290217, B290218

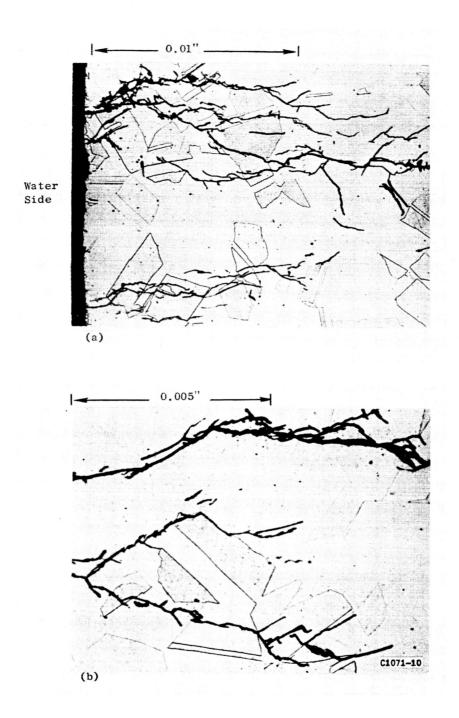


Figure 10. Photomicrographs of the Water Cooling Channel Wall at Two Magnifications. Note the Transgranular Nature of the Cracks Typical of Stress-Corrosion Cracking.

Etchant: 10g Oxalic Acid, 100cc Water, Electrolytic

- a) B290115, 250X
- b) B330112, 500X

TABLE III. ANALYSIS\* OF WATER SAMPLE TAKEN FROM COOLING CHANNEL EXIT LINE

	Chemical Analysis, ppm
Total hardness as CaCO3	130
Calcium as CaCO <sub>3</sub>	54
Magnesium as CaCO <sub>3</sub>	76
Carbonate as CaCO3	0
Bicarbonates as CaCO <sub>3</sub>	42
Chlorides as Cl	26
Chlorine as Cl <sub>2</sub>	0

<sup>\*</sup> pH: 8.15

Conductance:  $350 \times 10^{-6}$  mhos

the most significant aspect of the analytical results since the presence of chlorides and stress are normally the essential elements required to produce stress-corrosion cracking in stainless steel (4). (The referenced ASTM report is one of the most condensed and useful sources of information on the problem of stress-corrosion cracking and will be cited a number of times in this section). Although the chloride concentration is no doubt an important factor in most cases of stress-corrosion cracking, it should be pointed out that for some failures, "chlorides and stress were concluded to be the most likely cause of failure although sensitive chemical determinations did not disclose their presence (4)...

Several additional quotes from the ASTM report are felt to be relevant to the cracking of the cooling channel. "It is abundantly clear from this survey that no water of whatever degree of purity can be trusted as long as there is a mechanism whereby dissolved solids (chlorides) can be concentrated. There are numerous cases (of stress-corrosion cracking) involving only several ppm of chlorides. In the majority of these cases concentration has occurred by thermal means (5)."

Regarding the pH of the water, the ASTM reference reported incidents of stress-corrosion cracking for cases where the pH's varied from values of 2 to 14.

Samples of the upper cooling channel of the spool piece were analyzed to determine the carbon, hydrogen, nitrogen and oxygen concentrations of both the cracked and the adjacent uncracked areas and these results are given in Table IV. The carbon and sulfur concentrations were found to be below the specified maximum value (6) of 300 ppm. Spectrographic analysis confirmed that the channel material was of the 18-8 non-stabilized stainless steel type which includes Type 304L SS.

Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel

Stainless Steel, ASTM Special Technical Publication No. 264, Published by American Society for Testing Materials, March 1960, p. 6.

<sup>(5)</sup> Reference (4), p. 9.

<sup>(6)</sup> ASTM Designation A240-63.

## TABLE IV. CHEMICAL ANALYSES OF SAMPLES OF TYPE 304L

## SS\* FROM THE TOP COOLING CHANNEL OF

#### THE VACUUM CHAMBER SPOOL PIECE

	Chemical Analysis, ppm				
	<u>C</u>	H	N	0	S
Cracked sample	260, 290	10	459	313**	
Uncracked sample	210, 270	5	444	100	90

Type 304L SS was the specified material of construction and spectrographic analysis of samples of the cracked channel indicated that the material was this type of stainless steel.

Cracked surfaces slightly oxidized when heated during leak checking operations. This was probably responsible for the high oxygen concentration.

Hardness surveys were conducted on the metallographic specimens taken from the cracked region, B-1 in Figure 4, and the uncracked region, B-2, in Figure 4. The average of ten hardness impressions in the cracked region yielded a DPH\* value of 172. The average hardness value obtained in the uncracked specimen was DPH-151. These hardness values may be compared with handbook (7) values for Type 304 SS which indicated the following hardnesses in the various conditions: annealed-DPH-125, 10% cold work-DPH-160, and 20% cold work-DPH-205. This comparison suggests that both the upper portion (B-1) and the vertical portion (B-2) of the channel had the equivalent of approximately 10% cold work, which would be sufficient to contribute to stress-corrosion cracking if the other conditions, such as chloride and dissolved oxygen in the water, were also present. Although there was metallographic evidence of cold work in the structure of the channel in the bends (DPH-200), the structure of the specimens in the flat regions of the channel (DPH-151-172) showed little evidence of cold work (see Figures 9 and 10).

In an effort to determine if stress-corrosion cracking had occurred in the other cooling channels of the spool piece or in the channels of the other sections of the vacuum chamber shown in Figure 2, a number of cuts were made in selected locations. One of these test panels is shown in Figure 5. Three sides of the test panel were cut and the "flap" was then bent out approximately 75°. The inner surface was then examined visually for cracks. No cracks, pits or significant amounts of corrosion products were found. Additional test panels were cut and bent at location C in Figure 4, and in the top cooling channels of both the sump and the bell jar sections of the vacuum chamber. In all cases the channels were free of cracks and very similar in appearance to the middle channel of the spool piece described above.

5. <u>Discussion</u>. Review of the findings of this investigation indicates that stress-corrosion cracking of the stainless steel cooling channel was responsible for the chamber failure.

Diamond Pyramid Hardness, Load: 500 g.

<sup>(7)</sup> Metals Handbook, Vol. 1, 8th Edition, 1961, p. 544.

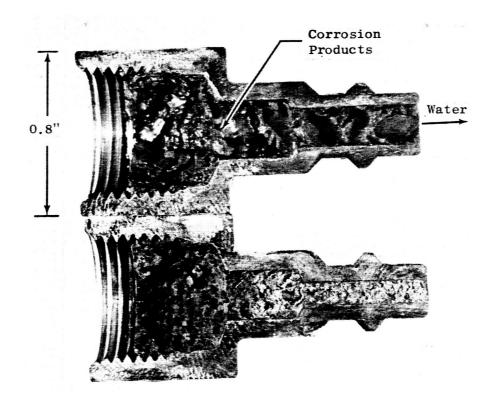
The critical factors associated with most of the stress-corrosion failures discussed in the ASTM report (4), i.e., fabrication and welding stresses, chlorides, and dissolved oxygen, were present in the chamber cooling channels. It seems most probable that the localized nature of the failure in the top half of the upper channel of the spool piece was caused by concentration of chlorides on the walls in this region. The water-line deposit shown in Figures 5, 6, and 8 indicates that this channel was only partially filled by the cooling water. The partially filled condition in this channel is attributed to: a) extensive blockage of the inlet and outlet water flow in the spool piece by the accumulation of corrosion products in the water-line fittings as illustrated in Figure 11, and b) the location of the water outlet fitting at the mid-point of the top channel. The location of the fitting at this point permitted the channel to drain to the approximate location of the water-line deposit with the low flow condition which resulted from the partial blockage of the restrictions in the inlet and outlet fittings.

The outlet temperature of the cooling water from the cracked channel did not exceed 120°F during the 3,809 hours of continuous test loop operation which preceded the failure. Although this would appear to be quite low for evaporation and concentration of chlorides, approximately 34% of the reported stress-corrosion failures (8) occurred with systems operating between 100°F and 200°F. It was also reported that a significant number of cases of stress-corrosion cracking have occurred at or near room temperature.

It is of interest to note that in the reported incidents of stress-corrosion cracking (9) 65% of the cases involved components containing stresses introduced by fabrication and welding. Stresses of both types were present in the region of most extensive cracking of the cooling channel. The hardness data obtained on the cooling channel specimens indicated that stress induced by cold work was certainly present.

<sup>(8)</sup> Reference 4, p. 11.

<sup>(9)</sup> Reference 4, p. 8.



C1071-11

Figure 11. Cadmium-Plated Steel Fitting Used to Connect Water Inlet and Outlet Lines to Spool Piece Cooling Channel. Both Inlet and Outlet Fittings Partially Plugged with Corrosion Products. (C66012124)

The phenomena responsible for the failure seems to be quite obvious at this time. The cracked channel has been isolated from the other channels and will be pumped by a vacuum system independent of the main chamber pumping system. The repaired channel and the turbomolecular pumping system which will be used to maintain the pressure in the "cracked" channel in the  $10^{-7}$  -  $10^{-8}$  torr range is shown in Figure 12.

Modifications of the chamber cooling system are planned following completion of the 5,000-hour test which will minimize the possibility of a re-occurrence of this problem. The modifications planned are listed below:

- a. The spool piece of the chamber in which the cracking of the cooling channels was observed will be rebuilt by the vendor using only the flanges from the cracked spool piece.
- b. The inlet and outlet fittings of the water cooling system on the spool piece will be located so as to assure that the channels are completely filled with water during test operation.
- c. Valves will be placed on the outlet side of each of the water cooling circuits.
- d. Stainless steel fittings will be used to replace the cadmium plated steel quick disconnect fittings in the water cooling circuits.
- e. Full flow filters will be placed in each of the cooling circuits to prevent particulate material from entering the cooling circuits.
- f. Low flow switches, will be placed in each of the three parallel circuits in the chamber cooling system. Low flow in any one of the circuits will activate an alarm on the control panel.
- g. The flow in each circuit will be measured every 100 hours to assure that low flow switches are operating properly and to detect any reduction in flow.

In summary, the cracking observed in the stainless steel water cooling channel described above is attributed to operation with a partially filled water cooling channel and the modifications and procedures described will prevent a re-occurrence of this in the future.

<sup>\*</sup> The removed segments of the channel were replaced with new stainless sheet and the remaining cracks were sealed with epoxy resin recommended for vacuum applications,

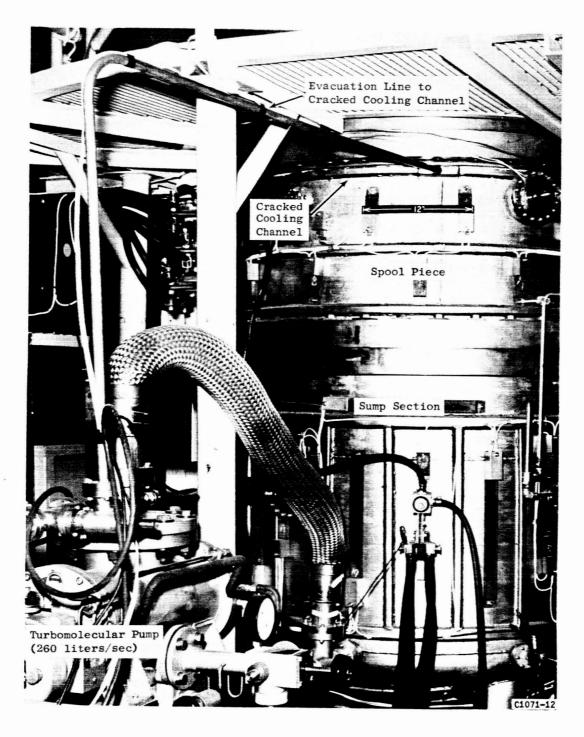


Figure 12. Spool Piece and Sump Section of the Prototype Corrosion Loop Test Chamber Showing the Evacuation Line and the Turbomolecular Pumping System Used to Pump Cracked Cooling Channel to a Pressure of  $10^{-8}$  Torr.

(C66031015)

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